WATERTOWN ARSENAL LABORATORIES

THE LATTICE PARAMETERS OF IRON-RUTHENIUM SOLID SOLUTION ALLOYS

TECHNICAL REPORT NO. WAL TR 830.2/1

BY

LEO J. COTTA, JR.
and
CHARLES P. GAZZARA

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GENERAL MATERIALS PROBLEMS, RESEARCH AND INVESTIGATION
D/A PROJECT 593-32-001

WATERTOWN ARSENAL
WATERTOWN 72, MASS.

AD

Lattice parameters
Iron alloys
X-ray diffraction

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TITLE

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ABSTRACT

The lattice parameters of iron-ruthenium solid solution alloys of 0.10, 0.20, 0.50, 0.75, 1.50, 2.25 and 3.00 weight percent ruthenium have been determined at 310 K using a Philips back-reflection focusing camera and annealed-powder specimens.

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INTRODUCTION

The measurement of lattice parameters of solids using X-ray diffraction cameras has been developed to the point where lattice parameters can be measured to an accuracy of 10^{-4} Å using standard cameras and techniques.[1,2,3]

The determination of the lattice parameters of the solid solution alloys of iron-ruthenium annealed powders with increasing ruthenium content to 3 weight percent ruthenium was performed using a standard back-reflection focusing camera (60 mm radius, Philips No. 52058). Raub and Plate[4], in their investigation of the iron-ruthenium phase diagram, have found the lattice parameters of two alloys up to 3 weight percent ruthenium. This data was of insufficient accuracy to be of use to these investigators.

In using the back-reflection focusing camera, a system utilizing an accurately notched film was worked out for calibrating the dimensions of the camera. Marking films for dimensional calibration is not new. This technique was used in 1933 by Owen and Iball.[5] The Philips camera has machined grooves, the positions of which are exposed on the film in recording a back-reflection exposure. These groove spacings should be calibrated for each camera. Therefore, a combination calibrated film notcher and cutter was used to accomplish this calibration.

In using Cohen's method of least squares[6], the error in the lattice parameter due to film shrinkage is eliminated, assuming a uniform film shrinkage. We have measured the nonuniformity in film shrinkage and corrected for this in our calculations of the lattice parameters.

PROCEDURE

Experimental

In order that the distance between grooves in the camera could be calibrated and to facilitate film cutting, a film cutter was constructed which cut the film into standard strips 12" x 1-3/8". A hole was punched in the center of the strips and 11 notches one inch apart were cut along the edge of the film (see Figure 2). The notch cutters were of ground tool steel accurately calibrated on both a traveling microscope and an optical comparator within 0.002 mm accuracy. To further check the notch dimensions and the stability of the notching cutters, several films and shim stock were cut in a constant temperature low-humidity room and measured (as were all subsequent exposed films) with a Gaertner optical comparator, which is accurate to 0.001 mm. However, the distance between notches can be measured to a limiting accuracy of 0.005 mm due to the rounding of the cut notches.

All films for this investigation were exposed at an ambient temperature of 310 ± 1/4 K. Therefore, the diameter of the cylindrical film holder of the camera was calibrated at 310 K using micrometers and standard gage blocks. During exposure the film temperature was maintained at 310 K by the arrangement shown in Figure 2. Two separate heaters were employed, one to maintain the temperature of the water surrounding the camera so that the temperature of the film holder was 310 K, and the other to preheat to 310 K the He gas flowing through the camera.

Unfiltered Fe radiation was used in order to obtain a sufficient number of diffracted lines. Two types of film were tested to determine uniformity of shrinkage and the one with the least shrinkage was selected for this investigation.

The iron which was used in making the alloys was 99.95+% pure with <0.009% carbon, <0.003% phosphorous, <0.016% sulphur, and <0.0005% nitrogen. The ruthenium was of 99.9+% purity. The Fe-Ru alloys were melted three times to insure homogeneity, machined clean of surface contamination, filed, and separated down to less than 44 microns. The powder specimens were placed in quartz capsules evacuated to 0.10 microns for 6 hours, sealed, and annealed. After annealing, each specimen was mounted and the remainder chemically analyzed.

Equations

The equations used in calculating the lattice parameter a. were developed by Cohen[6] and involve a least squares analysis:

$$A_o \sum_i a_i^2 + D\sum_i a_i \delta_i = \sum_i a_i \sin^2 \theta_i$$

$$A_o \sum_i a_i \delta_i + D\sum_i \delta_i^2 = \sum_i \delta_i \sin^2 \theta_i$$

where

$$A_o = \left(\frac{\lambda}{2a_o}\right)^2$$

$$\delta_i = 10\phi_i \sin 2\phi_i$$

λ = K_{α_1} wavelength (Å) = 1.93597Å

$\phi_i = 90° - \theta_i$

θ_i = Bragg angle for diffracted line i

$a_i = (h^2+k^2+\ell^2)$

D = drift constant

Applying the weighting factor $\text{cosec}^2 \Phi_i$ to the above equations, following Hess[7], in order to increase the weighting of the diffraction lines having θ values closer to 90 degrees yields:

$$A_o \sum_i \alpha_i^2 \text{cosec}^2 \Phi_i + D\sum_i \alpha_i \delta_i \text{cosec}^2 \Phi_i = \sum_i \alpha_i \tan^2\theta_i$$

$$A_o \sum_i \alpha_i \delta_i \text{cosec}^2 \Phi_i + D\sum_i \delta_i^2 \text{cosec}^2 \Phi_i = \sum_i \delta_i \tan^2\theta_i$$

RESULTS

The lattice constants were calculated using the above equations, correcting for the refractive index, and are listed below for each Fe-Ru alloy.

Wt.% Ru	Pure[8] Iron	Pure Iron Electrolytic	Pure Iron Carbonyl	0.10%	0.20%	0.50%	0.75%	1.50%	2.25%	3.00%
a_o(Å)	2.86678	2.8669_0	2.8665_7	2.8673_8	2.8680_3	2.8686_7	2.8688_6	2.8697_1	2.8706_2	2.8716_1

These lattice constants are plotted versus atomic percent Ru in Fe, see Figure 3, the diameter of the points corresponding to the approximate error in the determination of the lattice constants. The two filled-in bars in Figure 3 correspond to the values of the lattice parameters quoted by E. Raub and W. Plate[4], the height of the bars denoting the accuracy of measurement.

The average chemical composition of the alloys excluding Ru was essentially the same as given by the premelting analysis with the exception of carbon, which showed a slight increase in percentage, probably due to the filing operation. The analyzed Ru contents were, within experimental accuracy, identical to the calculated analyses based on the amount of Ru added.

The annealing time at 500 C required to begin recrystallization was determined experimentally for each Fe-Ru alloy, see Figure 4.

A comparison of two industrial films, A and B, tested for shrinkage, may be seen in Figure 5. Both the films illustrated were selected as typical examples. In general, Type A displayed a greater degree of shrinkage than Type B. It may also be seen from Figure 5 that Type B, which was used in this investigation, does change dimensionally during time intervals of the application of the film.

DISCUSSION

From the plot of lattice parameter versus atomic percent Ru (Figure 3) we see two points at 0% Ru. The hollow point was of a high purity iron specimen made from the same high purity iron used as a base material for the alloys. No deviation was made in specimen preparation. The filled-in point was from a pure carbonyl iron specimen which is available in powdered form, the classified portion having the same particle size as the other powder samples being used for X-ray examination. As may be seen from the tabulated values of the lattice parameters, the lattice parameter found by Sutton and Hume-Rothery[8] is slightly below that found for the pure filed iron (hollow point). The difference between the lattice parameters of the pure iron specimens experimentally found may be attributed to the slight carbon contamination due to filing the specimens. The solid line in Figure 4 is a plot of the reciprocal of the time to the beginning of recrystallization, (1/t) versus atomic percent Ru. The two broken lines approximate similar curves obtained by Leslie, et. al[9], where a change in slope for iron-manganese alloys was observed at approximately 0.3 atomic percent Mn. Such a doubly sloped relation seems to be consistent with the role of grain growth theory and impurity concentration in recrystallization. It would be worthwhile if such a relation could be calculated, whereby the annealing times could be determined for the preparation of powder samples for X-ray diffraction application.

The accuracy of determination of the lattice parameters, including error due to concentration variation, is approximately less than ± 0.0002 Å, the principal error being due to the limitations in measuring the peak position of the diffraction line (0.01 mm).

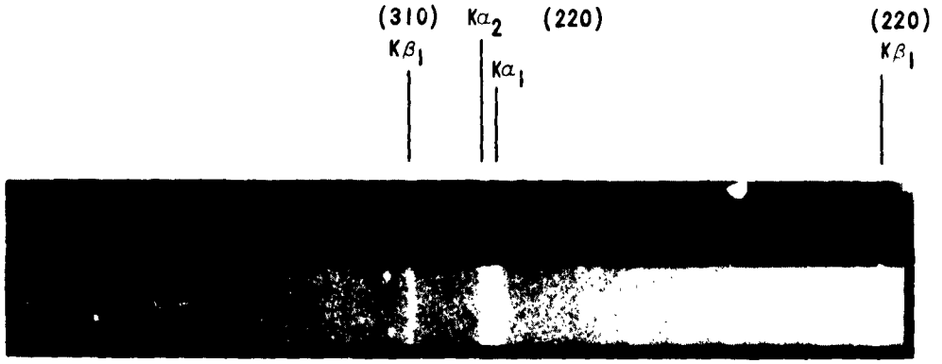
The choice of film for this application could have been either the Type A or the Type B and probably either film would have given similar results. This is primarily due to the fact that although Type A consistently displayed more shrinkage than Type B, the shrinkage of Type A was more uniform than that of Type B. In other words, the nonuniformity of both types of film was, on the average, the same.

SUMMARY

The lattice parameters of iron-ruthenium alloys up to 3.0 percent (by weight) ruthenium have been determined at a temperature of 310 K using a commercial back-reflection focusing camera allowing for nonuniform film shrinkage. The calculations were based on a Hess modification of Cohen's method of least squares.

ACKNOWLEDGMENT

The authors wish to thank Mr. W. Duffin who assisted in the preliminary experimental work on this project.



19-066-782/ORD-61

FIGURE 1: A TYPICAL BACK-REFLECTION FOCUSING PHOTOGRAPH (Fe-Ru (0.84 ATOMIC %) - ANNEALED @ 500 C FOR 35 HOURS)

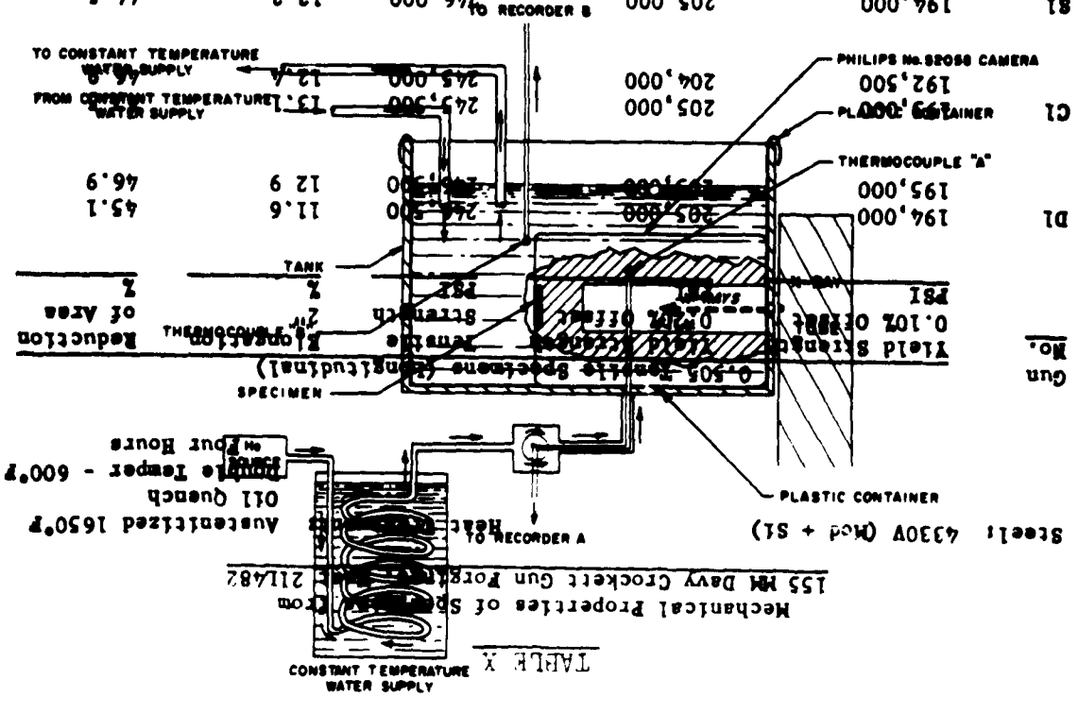
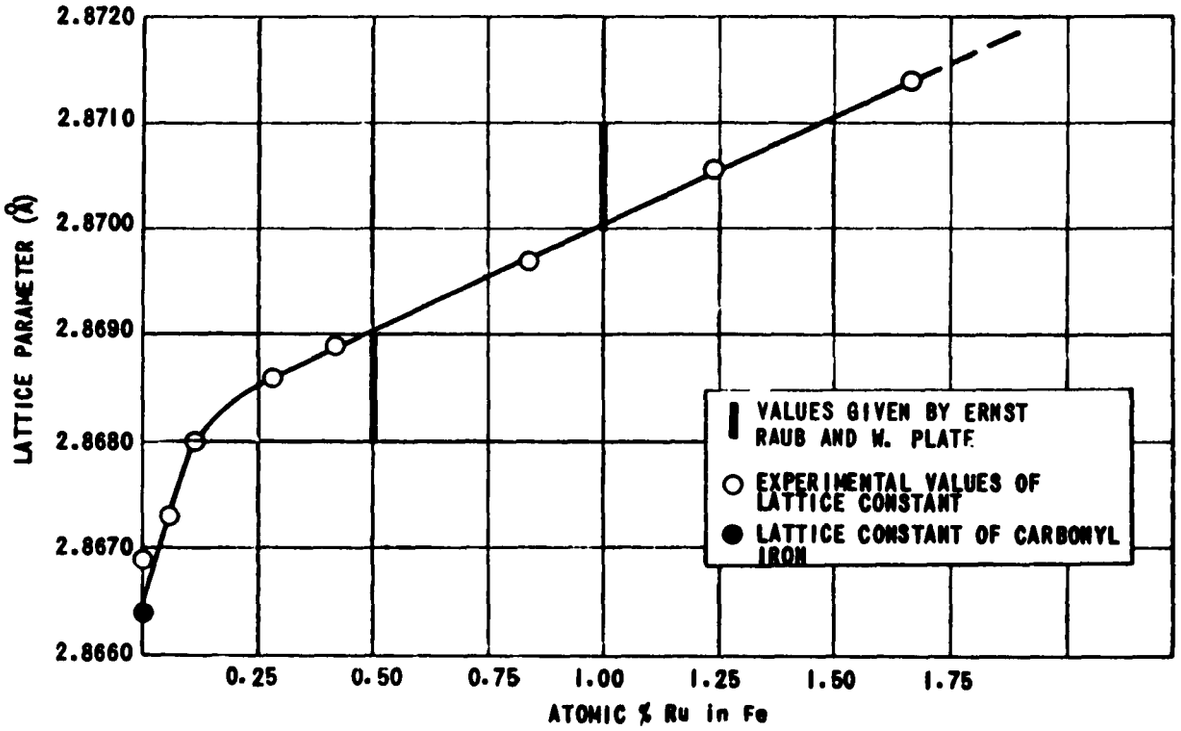


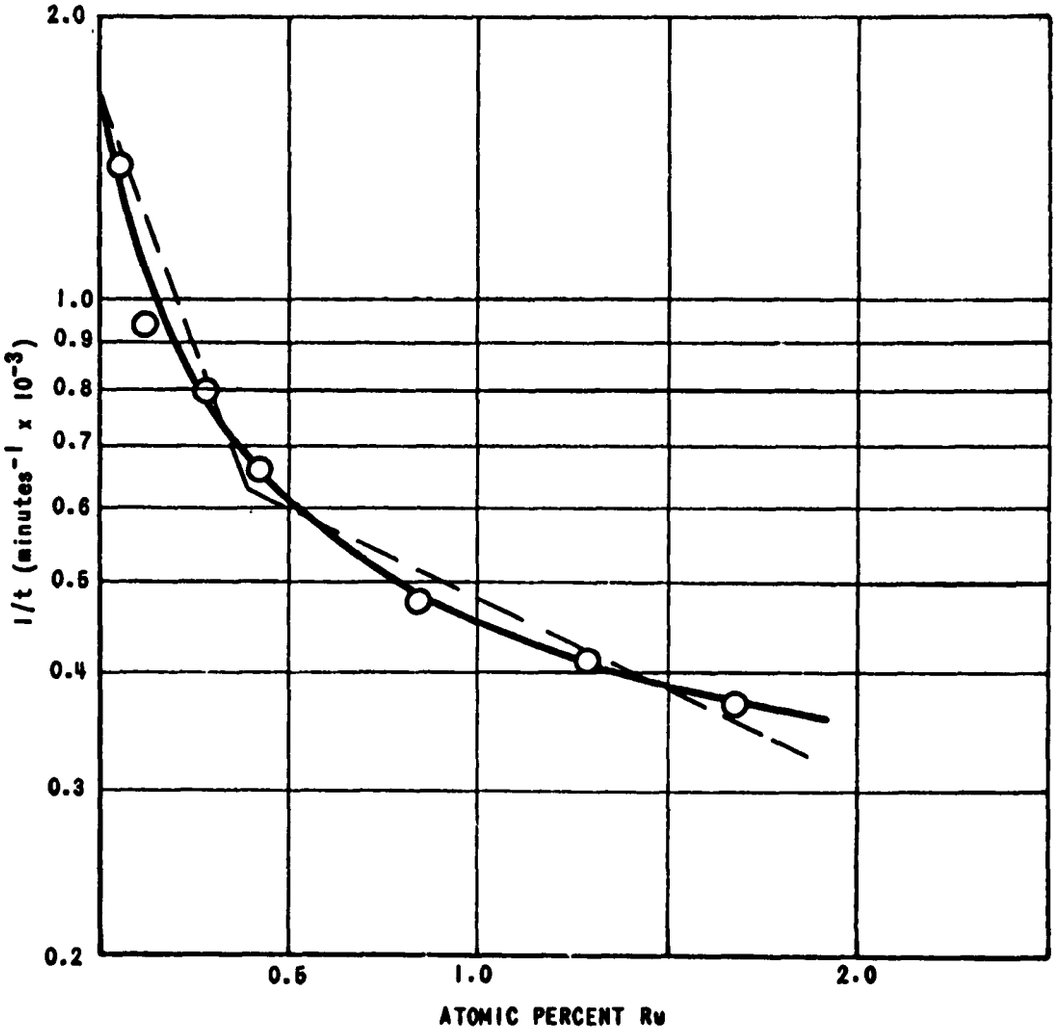
FIGURE 2: SCHEMATIC FOR X-RAY CAMERA

FIGURE 1 & 2



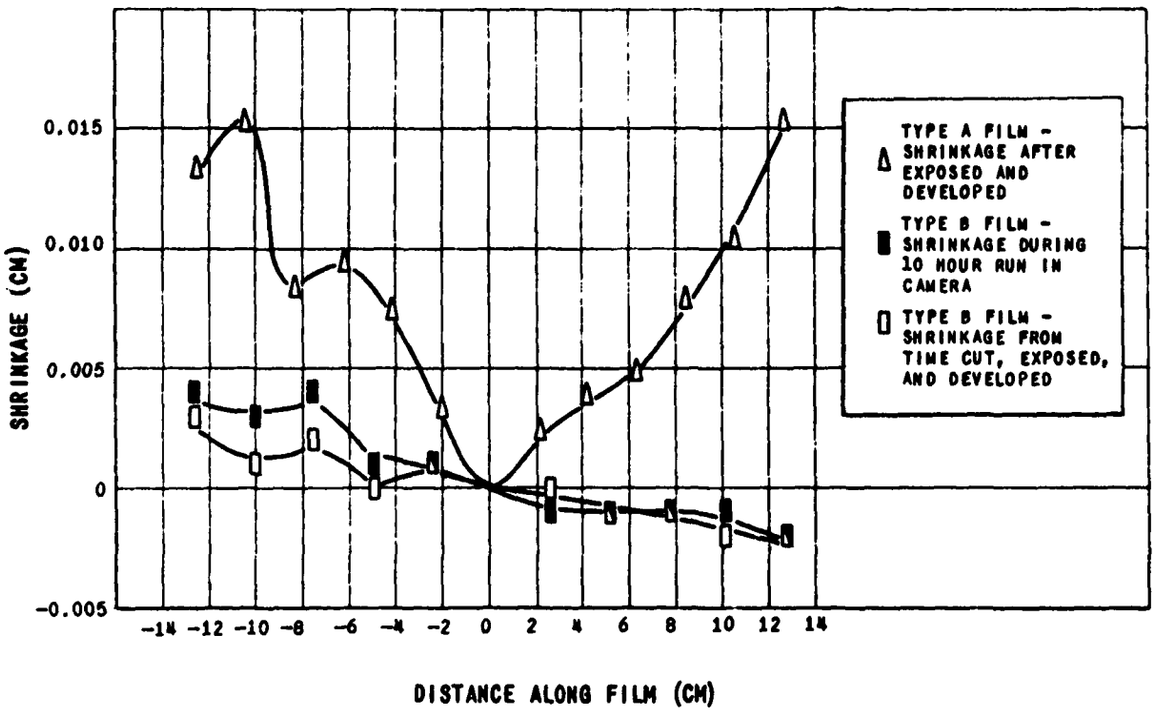
LATTICE PARAMETER VERSUS ATOMIC % Ru in Fe

FIGURE 3



RECIPROCAL OF TIME TO BEGIN RECRYSTALLIZATION
VS ATOMIC PERCENT Ru

FIGURE 4



SHRINKAGE OF TYPES A AND B FILMS

FIGURE 5

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